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AN EXPERIMENTAL INVESTIGATION OF THE RATE OF HYDROGEN ABSORPTION IN ZIRCONIUM-2.5 WT% NIOBIUM FROM STEAM/HYDROGEN MIXTURES AT ELEVATED TEMPERATURES
- TEST SPECIFICATIONS -

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by

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RESEARCH REPORT

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AN EXPERIMENTAL INVESTIGATION OF THE RATE OF HYDROGEN ABSORPTION IN ZIRCONIUM-2.5 WT% NIOBIUM FROM STEAM/HYDROGEN MIXTURES AT ELEVATED TEMPERATURES - TEST SPECIFICATIONS -

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ABSTRACT

Th[illegible]t specifications for an experimental program to study the r[illegible] of hydrogen absorption in zirconium-2.5 weight percent ni[illegible]m (Zr-2.5wt%Nb) pressure tube material from steam/hydrogen mixtures at elevated temperatures are discussed.

RÉSUMÉ

Le présent rapport traite des caractéristiques d'essais dans le cadre d'un programme expérimental sur l'absorption d'hydrogène par des tubes de force faits de zirconium contenant 2,5 pour cent de niobium en poids, dans un milieu de vapeur et d'hydrogène à hautes températures.

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-TEST SPECIFICATIONS-

A. INTRODUCTION

The test specifications for an experimental program to study the rate of hydrogen absorption in zirconium-2.5 weight percent niobium (Zr-2.5wt%Nb) pressure tube material from steam/hydrogen mixtures at elevated temperatures are discussed.

The test program objectives, the content of the test matrix and the rationale for the sequence of testing are discussed in this report. Suggestions are made with regards to the experimental setup and test procedures, and the post-test examination and data assessment that should be performed.

B. TEST PROGRAM OBJECTIVES

The objectives of the experimental investigation of the rate of hydrogen absorption in Zr-2.5wt%Nb from steam/hydrogen mixtures at elevated temperatures are to:

1. determine whether or not rapid hydrogen absorption into pressure tubes can occur under accident conditions of interest in CANDU reactors;

2. determine which factors have a major influence on the rate of hydrogen absorption into pressure tubes; and

3. determine whether rapid hydrogen absorption into pressure tubes occurs over a wide or narrow range of values for the parameters of interest.

The first objective addresses the question of whether or not the problem of rapid hydrogen absorption into pressure tubes under accident conditions in CANDU reactors exists. The last two objectives address the question 'If the problem exists, how extensive is it and what are the controlling factors?'.

Parameters which could influence the rate at which hydrogen is absorbed by pressure tubes include:

1. pressure tube temperature distribution;

2. time at temperature;

3. steam/hydrogen temperature;

4. mole per cent hydrogen in steam/hydrogen mixtures;

5. steam/hydrogen pressure;

6. steam/hydrogen flow velocity;

7. steam/hydrogen mass flow; and

8. the thickness and physical condition of the ZrO2 layer on the inner surface of the pressure tube.

The test matrix is designed to determine which of these parameters have the most influence on the rate of hydrogen absorption into pressure tubes.

C. CONTENT OF TEST MATRIX

The test conditions for the proposed hydrogen absorption experiments are given in Table 1. The rationale for the choice of conditions and sequence of testing is discussed in the next section. In defining the test matrix an attempt was made to minimise the number of tests required to meet the objectives described in Section B. Also, an attempt was made to select conditions of interest that would allow the design of the test apparatus to remain relatively simple and therefore not too expensive to construct. As an example, all tests are performed at atmospheric pressure.

D. TEST SEQUENCE AND RATIONALE

The rationale for the choice of test conditions and the sequence of tests is discussed in this section. The values of the parameters that appear in the proposed test matrix are summarised in Table 2. If all possible combinations of the parameter values were used then the number of tests would be 2,880. The proposed test matrix is comprised of 24 tests. An attempt is made, as early in the testing sequence as possible, to determine a set of conditions that results in rapid hydrogen absorption. The values of the important parameters are then varied, generally one at a time, from the rapid hydrogen absorption conditions in an attempt to scope the importance of the various parameters to the rate of hydrogen absorption. Thus, the results of these tests should indicate whether hydrogen absorption into Zr-2.5wt%Nb pressure tube material at elevated temperatures is possible and which parameters have the most influence on the rate of hydrogen absorption.

1. Test 1

The results from previous experimental work indicate that the conditions of Test 1 should result in rapid hydrogen absorption. The test specimen temperature is low enough (i.e. 800°C) that hydrogen generation due to the exothermic Zircaloy/steam reaction will be minimal when subsequent tests are performed at similar conditions to Test 1 but with steam present in the environment. Therefore, any hydrogen absorbed into the test specimen will originate from the environment. Also, since there is no initial zirconium dioxide (ZrO_2) layer on the surface of the test specimen, the rate of hydrogen absorption will not be affected by the time it would take the hydrogen to diffuse through a ZrO_2 layer. The pure hydrogen, near stagnant flow conditions of Test 1 (i.e. flow velocity = 0.0+ m/s) is a situation which is not expected to be highly probable under CANDU loss-of-coolant accident (LOCA) conditions. However, these conditions are the simplest to attain experimentally and should result in a measureable hydrogen absorption. As such they provide a good starting point for the test series since most variations of the parameters away from the Test 1 values should result in a decrease in hydrogen absorption.

2. Test 2

The only difference between Test 2 and Test 1 is that the flow velocity of the pure hydrogen environment has been increased from near zero to 0.5 m/s. A flow velocity of 0.5 m/s is at the low end of the conditions of interest to CANDU LOCA's as given in Table 1. However, for a pure hydrogen environment, flow velocity is not expected to greatly influence the timing and extent of hydrogen absorption. Thus, a flow velocity of 0.5 m/s is used since low velocities require the use of less hydrogen and are therefore simpler to attain experimentally. A comparison of the results from Tests 1 and 2 will give an indication of the effect of flow on the rate of hydrogen absorption for a pure hydrogen environment and a uniform test specimen temperature.

3. Test 3

The only difference between Test 3 and Test 1 is that the specimen temperature was uniform in Test 1 and in Test 3 there is a 400°C/m temperature gradient along the length of the specimen. Since the test specimens are 10.0 cm long the temperature difference between the ends of the specimen in Test 3 is 40°C. The inlet end is the hot end of the specimen. Hydrogen absorbed in the hotter region of the test specimen will move with time to the colder region of the test specimen. A comparison of the results from Tests 1 and 3 will give an indication of the effect of temperature gradient on the rate of hydrogen absorption for a stagnant, pure hydrogen environment.

4. Test 4

The changes in Tests 2 and 3 are combined in Test 4 to study the integrated effect of temperature gradient and flow velocity on the timing and extent of hydrogen absorption into the test specimen. Again, because of the pure hydrogen environment the effect of flow velocity on hydrogen absorption is expected to be minimal. Therefore, the results of Tests 3 and 4 should be similar. A comparison of the results of Tests 3 and 4 will give an indication of the effect of flow on the rate of hydrogen absorption for a pure hydrogen environment and a temperature gradient on the test specimen. A comparison of the results of Tests 2 and 4 will indicate the effect of temperature gradient on the rate of hydrogen absorption for a flowing, pure hydrogen environment.

5. Test 5

Test 5 will only be performed if the results of Test 1 through 4 inclusive indicate little or no hydrogen absorption during the 300 second test period. The conditions for Test 5 are those from the previous four tests which resulted in the most rapid hydrogen absorption. The time the test specimen is at temperature is increased to 600 seconds in Test 5.

6. Test 6

The environment in Test 6 is 50% hydrogen and 50% steam (i.e. hydrogen volume fraction in steam = 1.0) and the flow velocity is near zero. The temperature distribution of the test specimen is whatever resulted in the most hydrogen absorption in Tests 1 through 4 inclusive (i.e. uniform versus axial gradient). The time at temperature is 300 seconds if any of Tests 1 through 4 resulted in non-negligible hydrogen absorption. Otherwise the time at temperature is 600 seconds. The presence of steam in the environment is not expected to result in hydrogen generation due to the exothermic Zircaloy/steam reaction because the specimen temperature is too low (i.e. 800°C). The results of Test 6 will help to indicate the effect of the hydrogen volume fraction in a steam environment on the timing and extent of hydrogen absorption.

7. Test 7

The conditions for Test 7 are the same as for Test 6 except that the flow velocity is 0.5 m/s rather than near stagnant conditions. Flow velocity is expected to have an effect on the timing and extent of hydrogen absorption because the environment is no longer pure hydrogen. A flow velocity of 0.5 m/s will result in continued good mixing of the steam and hydrogen in the test chamber. As a result, there is no obvious need to do tests with higher flow velocities even in Tests where hydrogen generation will occur due to the exothermic Zircaloy/steam reaction on the test specimen. A comparison of the results of Tests 6 and 7 will give an indication of the effect of flow on the rate of hydrogen absorption for an hydrogen/steam environment.

8. Test 8

The conditions for Test 8 are those from Tests 1 through 7 inclusive that result in the greatest extent of hydrogen absorption with the exception that the test specimen temperature has been increased from 800°C to 1400°C. If the environment is not pure hydrogen then hydrogen generation will occur due to the exothermic Zircaloy/steam reaction on the test specimen. The results of Test 8 will give an indication of the effect of test specimen temperature on the rate of hydrogen absorption when compared to the results of the previous tests.

9. Test 9

The conditions for Test 9 are those from Tests 1 through 8 inclusive that result in the greatest extent of hydrogen absorption with the exception that the inlet steam/hydrogen temperature has been increased from 100°C to 800°C. If the environment is not pure hydrogen then hydrogen generation will occur due to the exothermic Zircaloy/steam reaction on the test specimen. Some of this hydrogen could be absorbed into the test specimen. The results of Test 9 will give an indication of the effect of environment temperature on the rate of hydrogen absorption when compared to the results of the previous tests.

10. Test 10

The conditions for Test 10 are those from Tests 1 through 9 inclusive that result in the greatest extent of hydrogen absorption with the exception that the test specimen has an initial layer of ZrO_2 present (i.e. 10 microns). It is expected that the presence of an initial, intact oxide layer will reduce the rate at which hydrogen is absorbed into the test specimen in comparison to the results of previous tests.

11. Test 11

Test 11 will only be performed if hydrogen absorption is rapid in Test 10 even though an initial, intact oxide layer of 10 microns is present on the absorbing surface of the test specimen. The conditions for Test 11 would be the same as for Test 10 except the initial, intact oxide layer thickness would be increased from 10 to 50 microns. The thicker, intact oxide layer is expected to reduce the rate of hydrogen absorption in comparison to the results from Test 10.

12. Test 12

Test 12 will only be performed if either Tests 10 or 11 result in negligible hydrogen absorption by the test specimen. The conditions for Test 12 will be the same as for Tests 10 and 11 except the time the test specimen is held at an elevated temperature would be increased. The initial oxide layer in Test 12 would be 10 microns thick if Test 10 resulted in negligible hydrogen absorption or 50 microns thick if Test 11 resulted in negligible hydrogen absorption. A longer time at temperature is expected to result in more extensive hydrogen absorption.

13. Test 13

Test 13 will only be performed if there is still negligible hydrogen absorption in Test 12. The Test 13 conditions will be the same as those for Test 12 except the time the test specimen is held at an elevated temperature would be increased.

14. Test 14

Test 14 will only be performed if there is negligible hydrogen absorption in Test 10 or 11. The conditions for Test 14 will be the same as for Tests 10 and 11 with the exception that the initial oxide layer would be cracked rather than intact. It is expected that a cracked oxide layer will offer less resistance to hydrogen absorption than an intact oxide layer. In Test 14 the initial oxide layer will be 10 microns thick if Test 10 showed negligible hydrogen absorption or 50 microns thick if only Test 11 showed little hydrogen absorption. The results from Test 14 will give an indication of the effect of oxide layer integrity on the rate of hydrogen absorption when compared to the results of previous tests.

15. Test 15

The conditions for Test 15 are those from Tests 1 through 9 inclusive that result in the greatest extent of hydrogen absorption with the exceptions that the flow velocity is 0.5 m/s and the hydrogen volume fraction in steam is 0.5. It is expected that hydrogen absorption will be less for reduced hydrogen volume fractions in steam. The results of Test 15 will give an indication of the effect of hydrogen volume fraction in steam on the rate of hydrogen absorption when compared to the results of previous tests.

16. Test 16

The conditions for Test 16 are identical to those for Test 15 with the exception that the hydrogen volume fraction in steam is increased from 0.5 to 5.0. The increase in hydrogen volume fraction in steam is expected to result in more extensive hydrogen absorption. The results of Test 16 will give an indication of the effect of hydrogen volume fraction in steam on the rate of hydrogen absorption when compared to the results of Test 15 and other previous tests.

17. Test 17

The conditions for Test 17 are the same as for Tests 15 and 16 with the exception that the specimen temperature distribution is no longer uniform but experiences an axial temperature gradient of 1000°C/m. As a result, the temperature difference between the two ends of the test specimen is 100°C. The hydrogen volume fraction in steam for Test 17 is that which results in the most hydrogen absorption from the previous tests. It is expected that the larger the axial temperature gradient the faster the diffusion of hydrogen from the hot region of the test specimen to the cold region of the specimen. The results of Test 17 will give an indication of the effect of test specimen temperature gradient on the rate of hydrogen absorption when compared to the results of previous tests.

18. Test 18

The conditions for Test 18 are the same as for Test 17 with the exception that the specimen temperature is reduced to 600°C. The temperature distribution on the test specimen is that which results in the most rapid hydrogen absorption from the results of the previous tests. It is expected that a reduction in the test specimen temperature may result in a decrease in the extent of hydrogen absorption. The results of Test 18 will give an indication of the effect of test specimen temperature on the rate of hydrogen absorption when compared to the results of previous tests.

19. Test 19

Test 19 will only be performed if the results of Test 18 indicate negligible hydrogen absorption. The conditions for Test 19 are identical to those of Test 18 with the exception that the time at temperature is increased.

20. Test 20

Test 20 will only be performed if the results of Test 19 indicate negligible hydrogen absorption. The conditions for Test 20 are identical to those of Test 19 with the exception that the time at temperature is increased.

21. Test 21

Test 21 will only be performed if the results of Test 18 indicate negligible hydrogen absorption. The conditions for Test 21 are identical to those of Test 18 with the exception that the gas environment is stagnant in Test 21. If the environment is pure hydrogen then the reduction in flow velocity is expected to have little effect on the extent of hydrogen absorption. If the environment is a steam/hydrogen mixture then a reduction in flow velocity may increase the rate of hydrogen absorption.

22. Test 22

Test 22 will only be performed if the results of Test 21 indicate negligible hydrogen absorption. The conditions for Test 22 are identical to those of Test 21 with the exception that the time at temperature is increased.

23. Test 23

The conditions for Test 23 will be those from Test 18 through 22 inclusive that resulted in the greatest amount of hydrogen absorption with the exception that there will be an initial, intact oxide layer 10 microns thick in Test 23. The results of Test 23 will give an indication of the effect of an intact oxide layer on the rate of hydrogen absorption for a reduced test specimen temperature when compared to the results of previous tests.

24. Test 24

Test 24 will only be performed if the results of Test 23 indicate negligible hydrogen absorption. The conditions for Test 24 are identical to those for Test 23 with the exception that the 10 micron oxide layer is cracked in Test 24. A cracked oxide layer is expected to allow more rapid hydrogen absorption than an intact oxide layer. The results of Test 24 will give an indication of the effect of oxide layer integrity on the rate of hydrogen absorption for a reduced test specimen temperature when compared to the results of Test 23.

E. EXPERIMENTAL SETUP AND TEST PROCEDURE

Descriptions of a proposed experimental setup and a procedure for testing are provided in this section. These are provided to give an indication of the overall requirements the experimental setup and testing procedure should attempt to meet. As such they should not be considered final designs but starting points for discussions which will result in the final designs. Also indications of the post-test examination and data assessment requirements are provided.

1. Description of Experimental Setup

A schematic of a preliminary experimental setup is shown in Figure 1. A schematic of a pressure tube specimen in the test chamber is shown in Figure 2.

It is proposed that the test chamber be a temperature resistant glass tube with an interior diameter of 2.5 cm. The test specimens are pieces cut from the longitudinal direction of an as-received zirconium-2.5wt% niobium pressure tube. The test specimens are 10.0 cm long, 1.0 cm wide and 0.42 cm thick. As a result, the flow cross-section in the test chamber with the test specimen in place would nominally be 4.5 cm2.

Hydrogen absorption under LOCA conditions can only potentially occur via the inner surface of the pressure tube. Thus, all but the "inner surface" of the test specimen should be sealed such that oxidation and hydrogen absorption cannot occur during a test. Each test specimen should be instrumented with two thermocouples located 1.0 cm from each end of the specimen (i.e. thermocouples T1 and T2 in Figures 1 and 2). The thermocouples should be imbedded approximately half way into the thickness of the test specimen. Two more thermocouples should be located 1.0 cm away from the ends of the test specimen to measure the "inlet" and "outlet" temperatures of the hydrogen/steam mixture around the test specimen (i.e. thermocouples T3 and T4 in Figures 1 and 2). Depending on the proposed method of heating the test specimen thermocouples T1 through T4 may have to be ungrounded.

As shown in Figure 1 the proposed experimental setup is such that two separate flow circuits are provided for the hydrogen and the steam. As a result the desired hydrogen and steam conditions can be established and measured prior to the initiation of a test without affecting the test specimen. Valves V1, V5, V9, V10, V11 and V12 are ordinary one-way valves. Valves V2, V6 and V13 are two-way valves. Valves V3 and V4 are identical, one-way valves that have a common adjustment. Valves V7 and V8 are identical, one-way valves that have a common adjustment.

A maximum volume of hydrogen of approximately 0.5 m3 has to be accommodated by the hydrogen collection/measurement system. This volume corresponds to pure hydrogen at a flow velocity of 0.5 m/s being supplied for 2400 seconds. The pre-mixing chamber has the same flow cross-section as the test chamber and will ensure complete mixing of hydrogen/steam mixtures prior to their entry into the test chamber. Provision should be made to potentially heat the hydrogen/steam mixtures in the pre-mixing chamber.

Enough instrumentation is proposed such that the following parameters can be well controlled at the initiation of, and during a test:

i) the temperature of the hydrogen/steam mixture around the test specimen;

ii) the flow velocity of the hydrogen/steam mixture past the test specimen; and

iii) the volume fraction of hydrogen to steam around the test specimen.

Provision should be made to measure the individual rates at which hydrogen and steam leave the test chamber. This will provide cross-checks on the measured amounts of hydrogen and steam flowing into the test chamber.

Care will have to be taken in the final design of the experimental setup to ensure there is as negligible a chance as feasible of forming explosive hydrogen/air mixtures. Examples of some of the precautions that could be provided are shown in Figure 1. An argon source is provided so that all circuits which experience hydrogen flow can be purged of air prior to, and after the introduction of hydrogen/steam mixtures. Oil traps are provided on the outlets of the circuits which experience hydrogen flow to prevent the backflow of air into these circuits. Provisions are made at the outlet of the circuits which experience hydrogen flow to vent the hydrogen to the atmosphere using argon as a carrier gas.

2. Test Procedure

The test specimen is cut from the longitudinal direction of an as-received Zr-2.5wt%Nb pressure tube and prepared as shown in Figure 3. Once the test specimen is prepared, instrumented and positioned in the test chamber and the flow circuit connections are completed. The test procedure is then dependent on whether the gas mixture is to be pure hydrogen, a mixture of hydrogen and steam or pure steam. The test procedures for pure hydrogen and hydrogen/steam mixtures are summarised in Tables 3 and 4 respectively.

3. Pre- and Post-test Examination

Prior to the performance of a test the following examination of the test specimen is completed:

i) the dimensions of the test specimen are measured at sample preparation stages a) through c) inclusive (refer to Figure 3);

ii) the test specimen is weighed at sample preparation stages a) through c) inclusive (refer to Figure 3);

iii) selected areas of the absorbing surface of the test specimen are photographed at approximately 50X - 100X magnification at sample preparation stage b) (refer to Figure 3);

iv) after sample preparation stage c) one excess end of the test specimen is mounted for macroscopic metallographic examination through the wall thickness. Photographs at approximately 50X - 100X magnification are taken of selected regions to characterise the pre-test hydride distribution in the test specimen and the oxide layer thickness and integrity on the absorbing surface of the test specimen; and

v) after sample preparation stage c) the other excess end of the test specimen is used to determine the initial hydrogen content of the test specimen.

After the completion of a test the following examination of the test specimen is performed:

i) the dimensions of the test specimen are measured;

ii) the test specimen is weighed;

iii) the test specimen is cut-up into 1.0 cm long pieces as shown in Figure 4;

iv) the test specimen pieces marked "ME" (refer to Figure 10) are used for macroscopic, metallographic examination to characterise the post-test hydride distribution and the physical characteristics of any ZrO2 layer present on the absorbing surface of the test specimen;

v) test specimen pieces "ME1" and "ME9" are used to examine the post-test condition of the imbedded portions of thermocouples T1 and T2; and

vi) the test specimen pieces marked "HC" (refer to Figure 4) are used to determine the post-test hydrogen content of the test specimen.

4. Data Assessment

In addition to the data available from the pre- and post-test examinations of the test specimens the following data will be available from each test:

i) the temperature history of the test specimen from thermocouples T1 and T2;

ii) the temperature histories of the gas mixture near the absorbing surface of the test specimen from thermocouples T3 and T4 (refer to Figures 1 and 2);

iii) the temperature and flow histories of the hydrogen entering the pre-mixing chamber from thermocouple T8 and flow meter F4 respectively (refer to Figure 1);

iv) the temperature and flow histories of the steam entering the pre-mixing chamber from thermocouple T11 and flow meter F7 respectively (refer to Figure 1);

v) the temperature and flow histories of the hydrogen/steam mixture entering the test chamber from thermocouple T5 and flow meter F1 respectively (refer to Figure 1);

vi) the rate at which steam leaves the test chamber from a measure of the rate at which condensate is collected (refer to Figure 1); and

vii) the rate at which hydrogen leaves the test chamber from a measure of the rate at which it is collected (refer to Figure 1).

Data iii) and iv) enable the volume fraction of hydrogen to steam to be determined throughout the test. Data v) and ii) enable the flow velocity in the test chamber to be determined throughout the test. These calculated values and the other measured data in conjunction with some of the pre-test examination results enable the determination of whether the desired test conditions were provided.

The post-test examination results will help define the range of conditions over which rapid hydrogen absorption into the test specimens can occur. Comparisons between test results as discussed in Section D will help determine which are the most important parameters that control the rate of hydrogen absorption into the test specimens.

Table 1-1
Test Conditions for Hydrogen Absorption Experiments
-Values in ( ) are those changed from the previous test-

TEST PARAMETER	TEST IDENTIFIER 1	2	3	4	5	6
SPECIMEN TEMPERATURE (°C)	800	800	800	800	800	800
TEMPERATURE GRADIENT (°C/m)	0	0	(400)	400	[2]	[2]
H2 VOLUME FRACTION IN STEAM	PURE H2	PURE H2	PURE H2	PURE H2	PURE H2	(1.0)
INITIAL ZRO2 THICKNESS (microns)	0	0	0	0	0	0
ZRO2 LAYER PHYSICAL CONDITION	NA	NA	NA	NA	NA	NA
TIME AT TEMPERATURE (seconds)	300	300	300	300	(600) [3]	(300) [4]
STEAM & H2 TEMPERATURE INLET (°C)	100	100	100	100	100	100
FLOW VELOCITY (m/s)	0.0+	(0.5)	(0.0+)	(0.5)	[2]	(0.0+)
INLET H2 & STEAM FLOW (g/s) [1]	NA	(0.018)	(NA)	(0.018)	[2]	(NA)

[NN] - Footnotes at end of table

Table 1-2
Test Conditions for Hydrogen Absorption Experiments
-Values in ( ) are those changed from the previous test-

TEST PARAMETER	TEST IDENTIFIER 7	8	9	10	11	12
SPECIMEN TEMPERATURE (°C)	800	(1400)	([7])	[8]	[8]	[8]
TEMPERATURE GRADIENT (°C/m)	[2]	([6])	([7])	[8]	[8]	[8]
H2 VOLUME FRACTION IN STEAM	1.0	([6])	([7])	[8]	[8]	[8]
INITIAL ZRO2 THICKNESS (microns)	0	0	0	(10)	(50) [9]	([10])
ZRO2 LAYER PHYSICAL CONDITION	NA	NA	NA	INTACT	INTACT	INTACT
TIME AT TEMPERATURE (seconds)	300 [4]	300 [4]	300 [4]	300 [4]	300 [4]	(600) [11]
STEAM & H2 TEMPERATURE INLET (°C)	100	100	(800)	[8]	[8]	[8]
FLOW VELOCITY (m/s)	(0.5)	([6])	([7])	[8]	[8]	[8]
INLET H2 & STEAM FLOW (g/s) [1]	([5])	([6])	([7])	[8]	[8]	[8]

[NN] - Footnotes at end of table

Table 1-3
Test Conditions for Hydrogen Absorption Experiments
-Values in ( ) are those changed from the previous test-

TEST PARAMETER	TEST IDENTIFIER 13	14	15	16	17	18
SPECIMEN TEMPERATURE (°C)	[8]	[8]	[8]	[8]	[8]	(600)
TEMPERATURE GRADIENT (°C/m)	[8]	[8]	[8]	[8]	(1000)	[17]
H2 VOLUME FRACTION IN STEAM	[8]	[8]	(0.5)	(5.0)	[16]	[16]
INITIAL ZRO2 THICKNESS (microns)	[10]	[13]	0	0	0	0
ZRO2 LAYER PHYSICAL CONDITION	INTACT	CRACKED	NA	NA	NA	NA
TIME AT TEMPERATURE (seconds)	(1200) [12]	300 [4]	300 [4]	300 [4]	300 [4]	300 [4]
STEAM & H2 TEMPERATURE INLET (°C)	[8]	[8]	[8]	[8]	[8]	[8]
FLOW VELOCITY (m/s)	[8]	[8]	(0.5)	0.5	0.5	0.5
INLET H2 & STEAM FLOW (g/s) [1]	[8]	[8]	[14]	[15]	[16]	[16]

[NN] - Footnotes at end of table

Table 1-4
Test Conditions for Hydrogen Absorption Experiments
-Values in ( ) are those changed from the previous test-

TEST PARAMETER	TEST IDENTIFIER 19	20	21	22	23	24
SPECIMEN TEMPERATURE (°C)	600	600	600	600	600	600
TEMPERATURE GRADIENT (°C/m)	[17]	[17]	[17]	[17]	[17]	[17]
H2 VOLUME FRACTION IN STEAM	[16]	[16]	[16]	[16]	[16]	[16]
INITIAL ZRO2 THICKNESS (microns)	0	0	0	0	(10)	10
ZRO2 LAYER PHYSICAL CONDITION	NA	NA	NA	NA	INTACT	CRACKED [23]
TIME AT TEMPERATURE (seconds)	(600) [18]	(1200) [19]	300 [4]	(1200) [21]	([22])	([22])
STEAM & H2 TEMPERATURE INLET (°C)	[8]	[8]	[8]	[8]	[8]	[8]
FLOW VELOCITY (m/s)	0.5	0.5	(0.0+) [20]	0.0+	([22])	([22])
INLET H2 & STEAM FLOW (g/s) [1]	[16]	[16]	(NA)	NA	([22])	([22])

[NN] - Footnotes at end of table

TABLE 1 FOOTNOTES

[1] Calculation of inlet H2 flow based on a cross-sectional flow area of 4.5 cm2 (i.e. test section diameter of 2.5 cm, sample cross-section of 0.42 cm2).

[2] Use the conditions from tests 1 through 4 inclusive (i.e. no gradient versus gradient, stagnant versus non-stagnant flow conditions) which resulted in the most rapid rate of hydrogen absorption.

[3] Test 5 will only be performed if the results of test 1 through 4 inclusive indicate little or no hydrogen absorption.

[4] Use 600s if test 5 was performed.

[5] Inlet H2 flow = 8.03(-3) g/s,
inlet steam flow = 7.23(-2) g/s,
where ZZ(-NN) is equivalent to ZZ x 10.$^{-NN}$

[6] Use conditions from tests 1 through 7 inclusive which resulted in the most rapid rate of hydrogen absorption.

[7] Use conditions from tests 1 through 8 inclusive which resulted in the most rapid rate of hydrogen absorption.

[8] Use conditions from tests 1 through 9 inclusive which resulted in the most rapid rate of hydrogen absorption.

[9] Test 11 will only be performed if hydrogen absorption is rapid in test 10.

TABLE 1 FOOTNOTES (CONT'D)

[10]Test 12 will only be performed if there is little or no hydrogen absorption in either test 10 or 11. The initial oxide thickness will be 10 microns if test 10 showed little or no hydrogen absorption or 50 microns if test 11 only showed little or no hydrogen absorption.

[11] Use 1200s if test 5 was required.

[12] Test 13 will only be performed if there is little or no hydrogen absorption in test 12. Use 2400s if test 5 was required.

[13] Test 14 will only be performed if there is little or no hydrogen absorption in test 10 or 11. The initial oxide thickness will be 10 microns if test 10 showed little or no hydrogen absorption or 50 microns if test 11 only showed little or no hydrogen absorption.

[14] Inlet H2 flow = 5.35(-3) g/s
Inlet steam flow = 9.55(-2) g/s

[15] Inlet H2 flow = 1.34(-2) g/s
Inlet steam flow = 2.41(-2) g/s

[16] Use the hydrogen to steam volume ratio which resulted in the most rapid hydrogen absorption in the previous tests.

[17] Use the temperature gradient which resulted in the most rapid hydrogen absorption in the previous tests.

[18] Test 19 will only be performed if the results of test 18 indicate little or no hydrogen absorption. Use 1200s if test 5 was required.

TABLE 1 FOOTNOTES (CONT'D)

[19] Test 20 will only be performed if the results of test 19 indicate little or no hydrogen absorption. Use 2400s if test 5 was required.

[20] Test 21 will only be performed if the results of test 18 indicate little or no hydrogen absorption.

[21] Test 22 will only be performed if the results of test 21 indicate little or no hydrogen absorption.

[22] Use the conditions which resulted in the most rapid hydrogen absorption in tests 18 to 22 inclusive.

[23] Test 24 will only be performed if the results of test 23 indicate little or no hydrogen absorption.

Table 2
Values of Parameters
in Proposed Test Matrix

Parameter	Values in Proposed Test Matrix			
Sample Temperature (°C)	600	800†	1200	
Sample Temperature Gradient (°C/m)	0	400	1000	
H2 Volume Fraction in Steam	PURE H2	5.0	1.0	0.5
Initial ZrO2 Thickness (microns)	0	10	50	
ZrO2 Layer Physical Condition	INTACT		CRACKED	
Time at Temperature (seconds)	300	600	1200	2400
Inlet Steam and Hydrogen Temperature (°C)	100	800		
Inlet Flow Velocity (m/s)	0+	0.5		

† NN - "Reference value"
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Table 3-1
Test Procedure - Pure Hydrogen
(Refer to Figure 1)

1. Ensure the steam supply circuit is "shutdown". Valve V5 should be closed and the two-way valve V6 should be in the position which isolates the steam circuit from the test circuit. Variable heater #2 should be shutoff. Valves 7 and 8 should be open.

2. The hydrogen and test circuits should be purged with argon prior to the introduction of hydrogen to either of these circuits. Valves V1 and V12 should be closed. Two-way valve V2 should be in the position which isolates the hydrogen circuit from the test circuit. Two-way valve V13 should be in the position which bypasses hydrogen collection/measurement system # 1 (which should be completely filled with distilled water) and allows the argon purge to pass through oil trap #1 to vent #1. Valves V3, V4 and V8 should be open and then valves V9, V10 and V11 can be opened to allow argon to flow into the hydrogen and test circuits.

3. Once the hydrogen circuit is purged close valve V9 and open valve V1. Adjust to the desired hydrogen flow and temperature as measured by flow meter F3 and thermocouple T7 respectively by regulating variable power heater #1 and the flow through valves V1 and V3. The hydrogen circuit is now ready to be valved into the test circuit.

4. Once the test circuit is purged close valve V11 and keep an argon flow in the test chamber by keeping valve V10 open. Heat the test specimen to the desired test temperature distribution. The test circuit is now ready for the introduction of hydrogen.

5. Close valve V10. The argon purge circuit is now isolated from all flow circuits.

6. Position two-way valve V13 such that flow through the test chamber will pass through hydrogen collection/measurement system #1. Immediately position two-way valve V2 such that the hydrogen source is connected to the test circuit.

Table 3-2
Test Procedure - Pure Hydrogen (cont'd)
(Refer to Figure 1)

7. If necessary, re-adjust to the desired hydrogen flow and temperature as measured by flow meter F1 and thermocouple T5 respectively by regulating flow with valves V1 and/or V4 and temperature with variable heater #1.

8. If necessary, re-adjust to the desired test specimen temperature distribution as measured by thermocouples T1 and T2 using the test specimen heater.

9. At the end of the test period close valve V1 and shutoff all power supplies. Position two-way valve V13 to bypass hydrogen collection/measurement system #1 and open valves V9 and then V11 to initiate argon purge of the test circuit. Open valve V3 to purge the remainder of the hydrogen circuit.

10. Take a gas sample from hydrogen collection/ measurement system #1. This will be used to determine the argon content in the collected gas such that the amount of hydrogen collected can be determined.

11. While the test circuit is still being actively purged with argon, position two-way valve V13 such that the argon purge gas passes through hydrogen collection/measurement system #1.

12. Periodically take gas samples from hydrogen collection/measurement system #1 to determine whether there is any remaining hydrogen. Once negligible hydrogen levels are attained valves V9 and V11 can be closed and hydrogen collection/ measurement system #1 refilled with distilled water.

Table 4-1
Test Procedure - Hydrogen/Steam Mixture
(Refer to Figure 1)

1. The hydrogen and steam circuits should be isolated from the test circuit. Two-way valve V2 should be in the position which isolates the hydrogen circuit from the test circuit. Two-way valve V6 should be in the position which isolates the steam circuit from the test circuit.

2. The hydrogen and test circuits should be purged with argon prior to the introduction of hydrogen to either of these circuits. Valves V1 and V12 should be closed. Two-way valve V2 should be in the position which isolates the hydrogen circuit from the test circuit. Two-way valve V13 should be in the position which bypasses hydrogen collection/measurement system # 1 (which should be completely filled with distilled water) and allows the argon purge to pass through oil trap #1 to vent #1. Valves V3, V4 and V8 should be open and then valves V9, V10 and V11 can be opened to allow argon to flow into the hydrogen and test circuits.

3. Once the hydrogen circuit is purged close valve V9 and open valve V1. Adjust to the desired hydrogen flow and temperature as measured by flow meter F3 and thermocouple T7 respectively by regulating variable power heater #1 and the flow through valves V1 and V3. The hydrogen circuit is now ready to be valved into the test circuit.

4. Adjust to the desired steam flow and temperature as measured by flow meter F6 and thermocouple T10 respectively by regulating variable power heater #2 and the flow through valves V5 and V7. The steam circuit is now ready to be valved into the test circuit.

5. Once the test circuit is purged close valve V11 and keep an argon flow in the test chamber by keeping valve V10 open. Heat the test specimen to the desired test temperature distribution. The test circuit is now ready for the introduction of hydrogen and steam.

Table 4-2
Test Procedure - Hydrogen/Steam Mixture (cont'd)
(Refer to Figure 1)

6. Close valve V10. The argon purge circuit is now isolated from all flow circuits.

7. Position two-way valve V13 such that flow through the test chamber will pass through hydrogen collection/measurement system #1. Open valve V12. Immediately position two-way valve V2 such that the hydrogen source is connected to the test circuit and then position two-way valve V6 such that the steam source is connected to the test circuit.

8. If necessary, re-adjust to the desired hydrogen flow and temperature as measured by flow meter F4 and thermocouple T8 respectively by regulating flow with valves V1 and/or V4 and temperature with variable heater #1.

9. If necessary, re-adjust to the desired steam flow and temperature as measured by flow meter F7 and thermocouple T11 respectively by regulating flow with valves V5 and/or V8 and temperature with variable heater #2.

10. If necessary, re-adjust to the desired test specimen temperature distribution as measured by thermocouples T1 and T2 using the test specimen heater.

11. At the end of the test period close valve V1. Position two-way valve V13 to bypass hydrogen collection/measurement system #1. Position two-way valve V6 such that the steam circuit is isolated from the test circuit. Open valves V9 and then V11 to initiate argon purge of the test circuit. Open valve V3 to purge the remainder of the hydrogen circuit. Shutoff all heaters.

12. Take a gas sample from hydrogen collection/ measurement system #1. This will be used to determine the argon content in the collected gas such that the amount of hydrogen collected can be determined.

Table 4-3
Test Procedure - Hydrogen/Steam Mixture (cont'd)
(Refer to Figure 1)

13. While the test circuit is still being actively purged with argon, position two-way valve V13 such that the argon purge gas passes through hydrogen collection/measurement system #1.

14. Periodically take gas samples from hydrogen collection/measurement system #1 to determine whether there is any remaining hydrogen. Once negligible hydrogen levels are attained valves V9 and V11 can be closed and hydrogen collection/measurement system #1 refilled with distilled water.

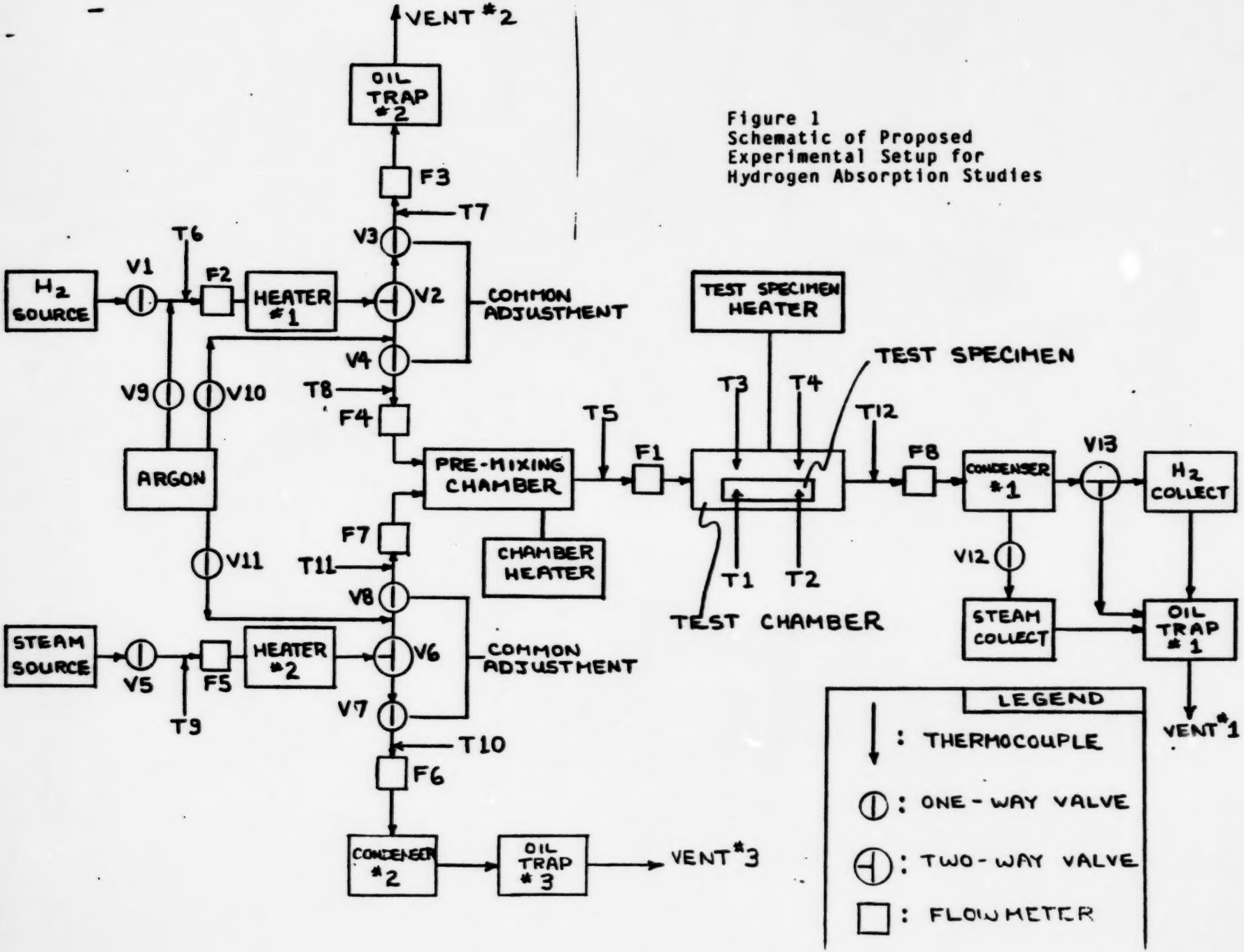

Figure 1
Schematic of Proposed
Experimental Setup for
Hydrogen Absorption Studies

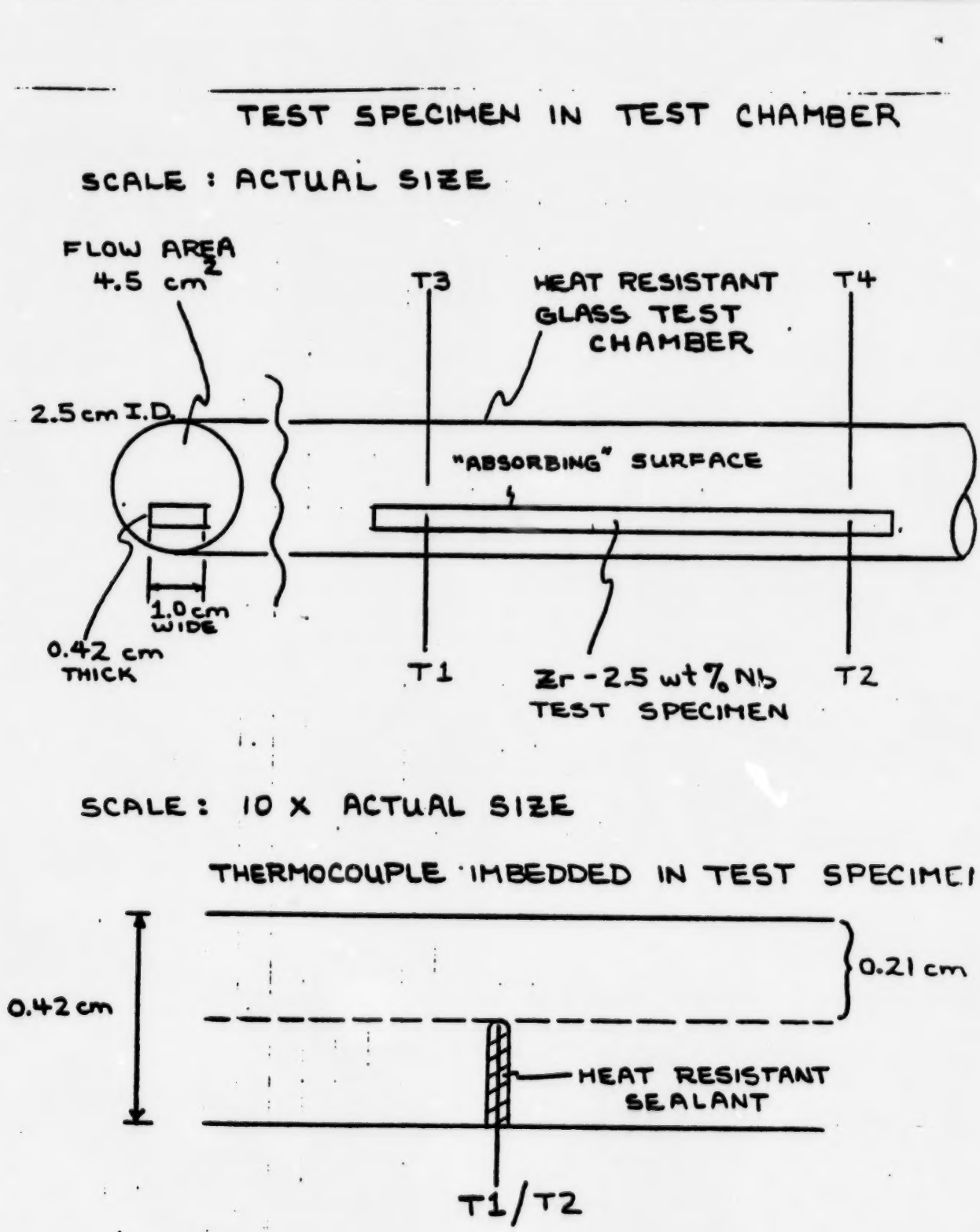

Figure 2
Schematic of Test
Specimen for
Hydrogen Absorption Studies

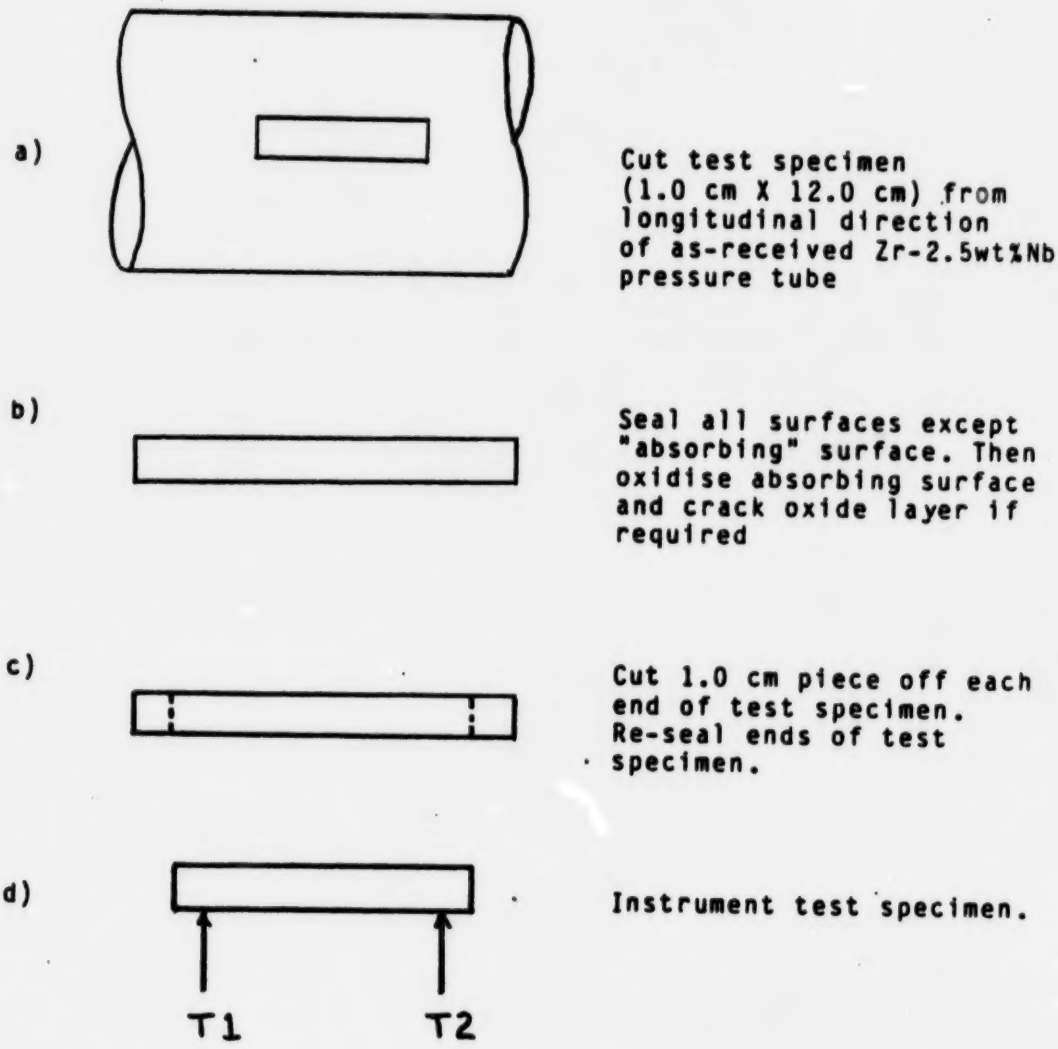
a)
Cut test specimen (1.0 cm X 12.0 cm) from longitudinal direction of as-received Zr-2.5wt%Nb pressure tube
b)
Seal all surfaces except "absorbing" surface. Then oxidise absorbing surface and crack oxide layer if required
c)
Cut 1.0 cm piece off each end of test specimen. Re-seal ends of test specimen.
d)
Instrument test specimen.
T1
T2

Figure 3
Test Specimen
Preparation Procedure

ME : SAMPLE FOR METALLOGRAPHIC EXAMINATION

HC : SAMPLE FOR HYDROGEN CONTENT ANALYSIS

Figure 4
Test Specimen Preparation
for Post-Test Examination